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AFRPL-TR-67-50

HALOGEN PASSIVATION STUDIES

Contract No. F04611-67-C-0033

W. A. Cannon, W. D. English, and N. A. Tiner

Astropower Laboratory, Missile & Space Systems Division
A Division of Douglas Aircraft Company, Inc.

TECHNICAL REPORT AFRPL-TR-67-50

February 1967

Air Force Rocket Propulsion Laboratory
Research and Technology Division
Air Force Systems Command
Edwards Air Force Base, California

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FOREWORD

This report was prepared by Astropower Laboratory, Advance Systems and Technology, Missile and Space Systems Division, Douglas Aircraft Company under Air Force Contract F04611-67-C-0033. The contract was administered by the Air Force Rocket Propulsion Laboratory, Research and Technology Division, Air Force Systems Command, Edwards Air Force Base, California, with Lt. Ralph Fargnoli as Project Engineer.

This report covers word done on the halogen passivation studies during the period from 1 November 1966 to 31 January 1967. The report was prepared by W. A. Cannon and W. D. English, under the supervision of N. A. Tiner.

Publication of this report does not constitute Air Force approval of the report's findings or conclusions. It is published only for the exchange and stimulation of ideas.

W. H. Ebelke, Colonel, USAF
Chief, Propellant Division

ABSTRACT

This progress report covers experimental work accomplished during the period from 1 November 1966 to 31 January 1967. During this time experiments were carried out to define requirements which must be met to remove organic contaminants from metal surfaces during passivation in fluorine. Metal coupons, porous stainless steel discs and glass capillaries were contaminated with various organic materials and exposed to gaseous fluorine and chlorine trifluoride vapor under a variety of conditions simulating typical passivation procedures. The conventional passivation procedures at room temperature and at moderate pressure are ineffective for removal of thin smears of organic contaminants from metal substrates.

TABLE OF CONTENTS

SECTION I

INTRODUCTION AND SUMMARY

This is the first of a series of quarterly reports covering work done from 1 November 1966 through 31 January 1967 on Contract No. F04611-67-C-0033. The major objectives of the subject contract are: (1) investigation of methods for producing the most suitable passive films on various metals; (2) testing effectiveness of the passivation procedure on system hardware components; and (3) preparing detailed procedures for obtaining the most durable passive surfaces.

The work has been divided into four phases for the purpose of pursuing the technical objectives of the contract. These phases are to be approached consecutively and are outlined in order as follows:

Phase I – Passive Surface Investigation
- a. Role of Contaminants
- b. Residual Fluorine
- c. Mechanical Stability

Phase II – Analysis of Passivation Methods

Phase III – Testing of Passivation Methods (developed in Phase II to demonstrate effectiveness, practicability and reliability)

Phase IV – Recapitulation of Passivation Methods (to prepare a design guide for simple systems)

The major technical effort described in the first quarterly report deals with Phase I investigations, primarily the role of contaminants.

Reactions of fluorine and chlorine trifluoride with gross potential system contaminants are described. The results to date have not been encouraging from the standpoint of relying on passivation for removal of final traces of contaminants. Fluorine and chlorine trifluoride in the concentrations proposed for passivation of metal surfaces react only slightly with typical organic contaminants when the latter are present as thin smears on a metal substrate.

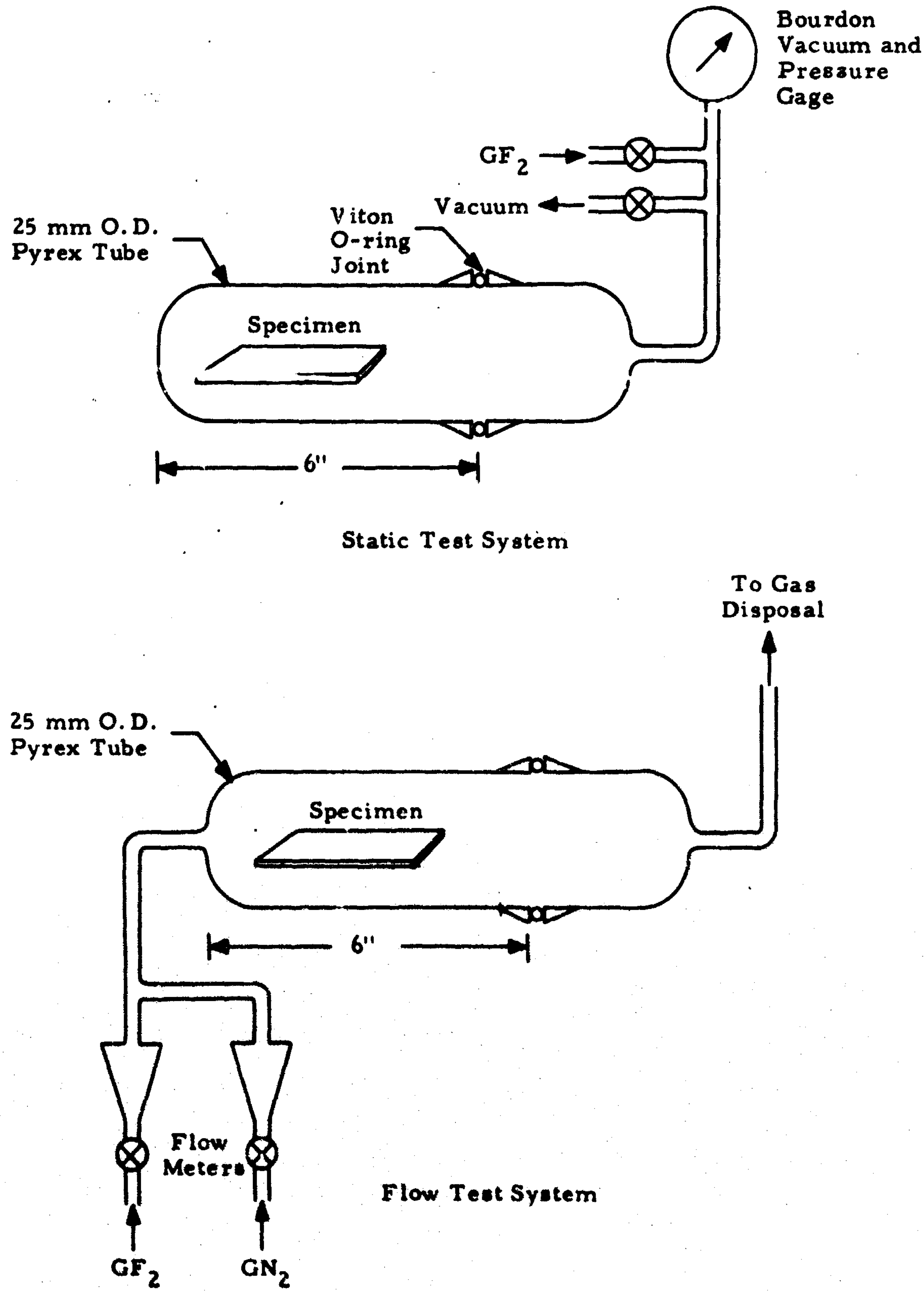

Figure 1. Static and Flow Systems for Exposure of Typical Contaminants to Fluorine Gas

SECTION II

EXPERIMENTAL

1. REACTION OF GASEOUS FLUORINE WITH POTENTIAL SYSTEM CONTAMINANTS

a. Apparatus and Procedures

The reactions of fluorine gas with typical system contaminants has been investigated as part of the Phase I studies dealing with the role of contaminants in the passivation process. It was desired, insofar as possible, to expose typical contaminants to fluorine in a manner simulating a passivation process. Two test methods were employed. The first method was a static fluorine exposure test in which the contaminant, smeared on a metal (nickel 200) coupon, was evacuated then exposed to fluorine in a closed system. A glass vessel was used so that reaction could be observed directly. The second test system had a double-ended glass tube through which either nitrogen or fluorine, or any desired mixture of the two gases, could be flowed continuously over the specimen. Schematics of the two systems are shown in Figure 1.

Two different modes of fluorine injection were used with each of the test systems. One mode used a slow, incremental buildup of fluorine pressure or concentration while the other provided a sudden application of the maximum fluorine pressure or concentration to the test specimen. The detailed procedures are described below.

(1) Static System – Incremental Pressure Buildup

With the specimen in place in the Pyrex tube, the system is first evacuated. Gaseous fluorine is admitted to a pressure of one psia for a period of five minutes, then the tube is again evacuated. The alternate five-minute pressurizations with gaseous fluorine and evacuations are continued at pressure levels of 2, 4, 8, and 15 psia. The specimen is kept under continuous observation from behind a safety shield during the exposure. Neglecting the time required to bring the pressures to the desired levels and for pumping out the fluorine between exposures, the specimens are exposed for a total of 25 minutes at gradually increasing fluorine pressure.

(2) Static System – Sudden Pressure Buildup

The contaminated specimen is first evacuated, then the system is abruptly pressurized to 15 psia with fluorine gas. After five minutes, the tube is pumped out. The specimen is kept under continuous observation.

(3) Flow System – Incremental Concentration Buildup

With the specimen in place in the Pyrex tube, a flow of gaseous nitrogen at a rate of 100 ml per minute is established through the tube. Gaseous fluorine is then added to the stream in flow increments of 10 ml/minute with five-minute intervals between increases until the flow rate of gaseous fluorine is 50 ml/minute. The flow rate of gaseous nitrogen is then decreased in decrements of 10 ml/minute until only pure fluorine is flowing in the system. After five minutes in pure fluorine the flow is shut off and the tube is purged with pure nitrogen. The specimen is observed frequently during the test. The total exposure time following this procedure is approximately 75 minutes with a gradually increasing partial pressure of fluorine.

(4) Flow System – Sudden Concentration Buildup

With the specimen in place in the tube, a nitrogen flow rate of 100 ml/minute is established. The flow of gaseous fluorine is abruptly increased to 50 ml/minute while the nitrogen flow rate is simultaneously decreased to zero. After five minutes the fluorine flow is stopped and the tube purged with pure nitrogen.

(5) Static System – High Pressure

For static test exposures of contaminated specimens at higher pressures, an all metal system was used. This was similar to the static system shown in Figure 1 except that the Pyrex tube was replaced with an equivalent section of one inch O.D. stainless steel tubing closed at one end and having an AN closure at the other end for connection to the gas manifold.

b. Contaminants

The following representative contaminants have been used in this phase of the investigation.

Vaseline – Petroleum Jelly U.S.P

KEL-F 90 – Halocarbon Stopcock Grease

Pydraul AC – Phosphate Type Industrial Hydraulic Fluid

Polyurethane – Polyurethane Foam Insulation

Acrylic Lacquer – Commercial Clear Lacquer in Aerosol Dispenser

Coupons 0.5" x 2" were cut from 0.025" nickel 200 sheet stock. A limited number of similar coupons of 2014 aluminum, 316 stainless steel, and Monel 400 were also used in this phase of the investigation. The edges of the coupons were filed to remove burrs. All coupons were acid etched and LOX cleaned to remove corrosion products and traces of oil and grease. Thin, uniform films of contaminants were applied individually to one side of separate coupons. Vaseline and KEL-F 90 were applied by smoothing on with glass rods. The Pydraul AC was applied as a small droplet from a

glass rod. It did not spread uniformly on the surface of substrate, however. The polyurethane foam was mixed and cured in place on the coupon. It does not foam uniformly due apparently to low temperature during cure. The acrylic lacquer was applied as a single wet coat from the aerosol dispenser. After final weighing the specimens were stored in a dust-free environment until ready for exposure.

Due to difference in film application the loading varied considerably among the various contaminants. In later experiments an effort was made to maintain the loading at a level near 50 mg per coupon. The calculated film thickness for vaseline at this loading is 4.5 mils.

c. Observations

Table I gives the data for a series of tests of contaminated nickel coupons exposed to fluorine gas in the static system at a maximum pressure of one atmosphere and at room temperature. Except for the KEL-F 90 specimens and one of the polyurethane-coated specimens, small weight gains were observed. None of the films ignited. Minor changes in appearance were observed only for vaseline and Pydraul AC while the others were unchanged.

Table II gives data for a similar series of experiments with the flow system. Similar results were obtained. Generally either weight gains or very small weight losses were observed.

Different alloys were used for coupons in the series of static system experiments in Table III. Only vaseline was used as the contaminant. All specimens except one lost weight, but the weight changes were small compared to the total amount of contaminant present.

In view of the apparent minor amount of reaction taking place in the experiments described above, contaminants were exposed to fluorine gas at 50 psig for one hour in a static system at room temperature. The data are presented in Table IV. Even at this pressure there was no ignition, but the relative weight gains of the vaseline film were greater. KEL-F 90-coated specimens lost weight.

These experiments confirm that fluorine gas under conditions of pressure, concentration and temperature frequently employed for system passivation, is relatively ineffective for reacting with and removing typical organic system contaminants. Although some materials do undergo partial reaction, as evidenced by weight changes and alterations of appearance, the reactions appear to be limited largely to fluorination reactions rather than complete reaction to volatile reaction products.

TABLE I. EXPOSURE OF CONTAMINATED SPECIMENS (0.5" x 2") TO FLUORINE GAS — STATIC SYSTEM

Specimen Number	Contaminant	Uncoated Specimen Weight	Weight of Contaminant	Wt. Change During Exposure	Remarks
A. Incremental Pressure Buildup					
Nickel N8	Vaseline	3.2158 g	0.0061 g	+ 0.0008 g	Film became partly opaque
Nickel N11	KEL-F 90	3.2290 g	0.0227 g	- 0.0008 g	No visible change
Nickel N41	Pydraul AC	3.2842 g	0.0058 g	+ 0.0004 g	Droplets became cloudy
Nickel N36	Polyurethane	3.1942 g	0.0679 g	+ 0.0015 g	No visible change
Nickel N34	Acrylic Lacquer	3.2799 g	0.0029 g	+ 0.0004 g	No visible change
B. Sudden Pressure Buildup					
Nickel N11	Vaseline	3.0917 g	0.0092 g	+ 0.0006 g	Film became partly opaque
Nickel N18	KEL-F 90	3.2616 g	0.0242 g	- 0.0014 g	No visible change
Nickel N12	Pydraul AC	3.1195 g	0.0049 g	+ 0.0005 g	Droplets became cloudy
Nickel N30	Polyurethane	3.1933 g	0.0394 g	- 0.0008 g	No visible change
Nickel N24	Acrylic Lacquer	3.2678 g	0.0029 g	+ 0.0002 g	No visible change

TABLE II. EXPOSURE OF CONTAMINATED NICKEL SPECIMENS (0.5" x 2") TO FLUORINE GAS – FLOW SYSTEM

Specimen Number	Contaminant	Uncoated Specimen Weight	Wt. of Contaminant	Wt. Change During Exposure	Remarks
A. Incremental Concentration Buildup					
Nickel N33	Vaseline	3.3205 g	0.0394 g	+ 0.0033 g	Film became cloudy
Nickel N26	KEL-F 90	3.1580 g	0.0187 g	0.0000	None
Nickel N28	Pydraul AC	3.1623 g	0.0287 g	+ 0.0048 g	Liquid appeared to partially solidify
Nickel N1	Polyurethane	3.0573 g	0.0490 g	+ 0.0031 g	None
Nickel N21	Acrylic Lacquer	3.4236 g	0.0236 g	+ 0.0035 g	None
B. Sudden Concentration Buildup					
Nickel N15	Vaseline	3.2041 g	0.0581 g	- 0.0033 g	Film became cloudy
Nickel N29	KEL-F 90	3.1705 g	0.0153 g	- 0.0001 g	None
Nickel N38	Pydraul AC	3.0686 g	0.0504 g	- 0.0002 g	Liquid partially solidified
Nickel N31	Polyurethane	3.0872 g	0.0367 g	+ 0.0004 g	Duller appearance
Nickel N21	Acrylic Lacquer	3.1427 g	0.0220 g	+ 0.0006 g	None

TABLE III. EXPOSURE OF CONTAMINATED SPECIMENS (0.5" x 2") TO FLUORINE GAS – STATIC SYSTEM – CONTAMINANT VASELINE

Specimen Composition and Number	Uncoated Specimen Weight	Weight Contaminant	Weight Change During Exposure	Remarks
A. Incremental Pressure Buildup				
2014 Aluminum (B1)	1.7398	0.0537	- 0.0014	Turned cloudy and small bubbles formed
316 SS (J1)	2.1339	0.0536	+ 0.0004	Turned cloudy
Monel 400 (Q1)	3.5070	0.0493	- 0.0020	Turned cloudy
B. Sudden Pressure Buildup				
2014 Aluminum (B2)	1.7440	0.0501	-0.0019	Turned cloudy
316 SS (J2)	2.2275	0.0564	-0.0009	Turned cloudy
Monel 400 (Q2)	3.5313	0.0498	-0.0025	Turned cloudy

TABLE IV. EXPOSURE OF CONTAMINATED NICKEL SPECIMENS (0.5" x 2")
50 PSIG FLUORINE GAS — ONE HOUR — STATIC SYSTEM

Specimen Number	Contaminant	Uncoated Specimen Weight	Weight of Contaminant	Weight Change During Exposure
Nickel N3	Vaseline	3.2352	0.0490	+ 0.0086
Nickel N4	Vaseline	3.1825	0.0511	+ 0.0094
Nickel N5	KEL-F 90	3.1368	0.0484	- 0.0019
Nickel N7	KEL-F 90	3.1613	0.0587	- 0.0004

2. REACTION OF CHLORINE TRIFLUORIDE WITH SYSTEM CONTAMINANTS

a. Apparatus and Procedure

The static system (Figure 1) was used and chlorine trifluoride vapor was introduced abruptly to the evacuated system containing the contaminated coupon. A maximum pressure of one atmosphere was used with the system initially at room temperature.

b. Observations

The results for vaseline and KEL-F 90 contaminated nickel 200 coupons are given in Table V. The vaseline film ignited in chlorine trifluoride proving that this medium is more reactive than fluorine at equivalent pressure. However, the residue left weighed approximately 50 per cent more than the original vaseline film. The KEL-F 90 film liquified and bubbled. This behavior is attributed to the reaction of the chlorine trifluoride with the finely-divided silica used as a thickener in the grease, to form silicon tetrafluoride.

3. REACTION OF GASEOUS FLUORINE WITH CONTAMINATED POROUS STAINLESS STEEL

a. Apparatus and Procedures

Filtermet stainless steel (type 316) filter elements in disc form 1/16" thick and 1/2" diameter were impregnated with Halocarbon oil and dye penetrant fluid. Samples covering a range of porosities from about 30 to 50 per cent were used. Mean pore sizes were on the order of 30 microns.

Impregnation was carried out by immersing the discs in the respective fluid, heating under vacuum to about 50° - 60°C to remove air from pores, then releasing the vacuum and cooling the specimens under the impregnating fluid. Finally the specimens were removed from the fluid and the surfaces superficially dried by blotting with absorbent paper. The impregnated specimens were tested for reaction with fluorine by exposure in the static test system to fluorine gas at one atmosphere for one hour at room temperature.

b. Contaminant Fluids

Halocarbon Oil — Series 13-21 Halocarbon Products Corporation.

Dye Penetrant Fluid — Contrast Dye Penetrant-MIL-L-25135, Group 1. This material partially volatilized during the vacuum impregnation of the porous stainless steel discs.

TABLE V. EXPOSURE OF CONTAMINATED NICKEL SPECIMENS TO CHLORINE TRIFLUORIDE VAPOR – ONE ATMOSPHERE – STATIC SYSTEM

Substrate	Contaminant	Uncoated Specimen Weight	Weight of Contaminant	Wt. Change During Experiment	Remarks
Nickel N10	Vaseline	3.1675 g	0.0567 g	+ 0.0263 g	Sample ignited in 30 seconds. Flame died out and exposure continued for one hour. Black, waxv residue left on speciment.
Nickel N6	KEL-F 90	3.0466 g	0.0495 g	- 0.0015 g	Grease liquified and small bubbles appeared in film after three minutes. Exposure continued for one hour.

c. Observations

The results of the exposure of the contaminated specimens to fluorine gas are given in Table VI. The Halocarbon impregnated specimens were unchanged in weight or appearance. The Dye Penetrant-impregnated specimens burst into flame upon initial pressurization with fluorine. The flame subsided after a few seconds after which pressurization with fluorine was resumed and exposure continued for one hour. The weight losses of the specimens ranged from 10 to nearly 50 per cent of the original weight of contaminant.

4. REACTION OF GASEOUS FLUORINE WITH DYE PENETRANT FLUID IN GLASS CAPILLARIES

a. Apparatus and Procedure

Two Pyrex glass capillaries, one 100 micron and the other 30 micron inside diameter were filled with Dye Penetrant fluid. One inch sections of each capillary were broken out and the filled capillaries exposed individually in flowing fluorine gas in the flow system (see Figure 1). The tubes were kept under continuous observation with a binocular microscope during the exposure.

b. Contaminant

Dye Penetrant Solution — Contrast Dye Penetrant-MIL-L-25135, Group 1.

c. Observations

After about five minutes exposure the dye penetrant fluid in the 100 micron capillary turned black or dark blue at the meniscus. This change from the initial red color penetrated further into the fluid in the capillary to a depth of about 400 microns after one hour. The meniscus was originally 700 microns inside the capillary and did not recede further during the exposure of one hour. In view of these observations it seems that only a very slow reaction takes place due to slow diffusion into and out of the capillary.

The observations with the 30 micron capillary were similar except the depth of discoloration in the fluid was only about 100 microns.

5. PRESENCE OF RESIDUAL OILS IN ROLLED ALUMINUM SHEET STOCK

It has been observed in previous activities unrelated to this project that some commercial aluminum and copper foils have oils or fatty acids rolled into fissures or surface defects and that these materials are not readily removed by any conventional degreasing process. The contaminants frequently reveal themselves during vacuum work with the foils at which time it is found that surfaces are slow to outgas under high vacuum. When heated

TABLE VI. EXPOSURE OF POROUS STAINLESS STEEL SPECIMENS TO FLUORINE – ONE ATMOSPHERE – ONE HOUR – STATIC SYSTEM

Specimen	Contaminant	Weight Specimen	Weight Contaminant	Weight Change	Remarks
316SS - 32% porosity	Halocarbon Oil	1.1415 g	0.1159 g	+ 0.0002 g	No visible reaction
316SS - 41% porosity	Halocarbon Oil	0.9711 g	0.1280 g	+ 0.0006 g	No visible reaction
316SS - 50% porosity	Halocarbon Oil	0.8556 g	0.1638 g	- 0.0002 g	No visible reaction
316SS - 32% porosity	Dye Penetrant	1.1206 g	0.0536 g	- 0.0190 g	Burst into flame on initial exposure. After flame died out exposure continued for one hour total.
316SS - 41% porosity	Dye Penetrant	0.9745 g	0.0712 g	- 0.0071 g	
316SS - 50% porosity	Dye Penetrant	0.7877 g	0.0792 g	- 0.0347 g	

to several hundred degrees in vacuum the foils may release considerable amounts of fatty acids which condense in cooler parts of the vacuum system. The implication of these observations is that during the rolling process, a fatty acid, probably stearic acid, is used to lubricate the rolls and that this material is forced under high pressure into minute fissures in the surface. The passageways connecting these fissures with the surface are either partially closed off or are of such small dimensions that normal cleaning and degreasing operations are inefficient.

A total of 50 specimens 1/2" x 4" were sheared from 2014 aluminum sheet stock. These were LOX cleaned by a conventional technique described as follows:

Dye wiped off with acetone

Oakite dip for 30 seconds, followed by immediate water flush

Dried in nitrogen

Vapor degreased in trichlorethylene

Dipped in Freon

Dried in nitrogen

Following the cleaning process the strips were enclosed in plastic bags and were handled only with degreased forceps.

The degreased strips (total area 200 square inches) were sealed into a 30 mm OD Pyrex tube which in turn was connected to a high vacuum system with a 5 mm Pyrex tube. The large tube was heated slowly with a tube furnace to 300°C while under a vacuum of 10^{-5} torr or lower. As the temperature approached 200°C a slight fogging inside the 5 mm connecting tube was noted at a point immediately outside the tube furnace. The fogging increased until finally discrete, oily droplets could be observed. When no further increase in volume of the oil could be detected, the tube was cooled while still under vacuum. Finally the section of the connecting tube containing the oil was cut out. The total quantity of molecularly distilled oil was estimated to be about 1 milligram. It was identified from its infrared spectrum as a fatty acid.

Identical experiments were carried out with aluminum 2024 and nickel 200 specimens. The results were completely negative, i.e., no trace of material was molecularly distilled from the materials.

Within the limited scope of the experiments, it seems that either no contamination problem exists with commercial sheet stock used in this project or that the problem is minor. It is probable that one milligram of contaminant per 200 square inches of sheet stock represents an insignificant level of contamination. If any problem arises in this regard, it should be a simple manner to remove most or all of any contamination of this type by a mild etching of the sheet stock prior to degreasing.

SECTION III

FUTURE WORK

The projected Phase I studies to be completed in the next quarterly period are outlined as follows.

1. ROLE OF CONTAMINANTS

Additional work will include impact testing of contaminated porous stainless steel and aluminum test coupons. The contaminated porous bodies will be exposed to various passivation cycles, then will be subjected to impact tests in the ABMA drop weight tester in liquid fluorine.

Surface contaminated coupons will be subjected to various passivation procedures and impact tested as described above.

2. EFFECT OF RESIDUAL FLUORINE

Work will be initiated on the effects of residual fluorine on corrosion of metal surfaces.

3. MECHANICAL STABILITY TESTS

The flexure tests for passivated bellows specimens will be completed during the next quarterly period.

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13. ABSTRACT

This progress report covers experimental work accomplished during the period from 1 November 1966 to 31 January 1967. During this time experiments were carried out to define requirements which must be met to remove organic contaminants from metal surfaces during passivation in fluorine. Metal coupons, porous stainless steel discs and glass capillaries were contaminated with various organic materials and exposed to gaseous fluorine and chlorine trifluoride vapor under a variety of conditions simulating typical passivation procedures. The conventional passivation procedures at room temperature and at moderate pressure are ineffective for removal of thin smears of organic contaminants from metal substrates.

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Stainless Steel						
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